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~~PROGRESS~~ REPORT

Final

For the Period of
March 1, 1998 to July 31, 1998

GRANT:
Biodegradation of Polymeric Coatings and Corrosion of Aircraft Materials

GRANTEE:
University of Dayton
300 College Park Avenue
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GRANT NO.:
F49620-98-1-0297

PRINCIPAL INVESTIGATOR:
Kimberly A. Trick Ph.D.

OBJECTIVES

Atmospheric chemical corrosion is a severe threat to metal aircraft structures in their working environment. Previous wovrk[1] has suggested that the presence of biological activity can enhance the development of corrosion of an aluminum alloy coated with an organic primer. The objective of the current work is to further verify that corrosion of aluminum alloys is microbially enhanced and to investigate the mechanisms of the contribution of microorganisms to degradation of coatings and corrosion of aluminum.

Currently the aerospace industry utilizes chromates added to organic coatings to inhibit corrosion of aluminum alloys. Chromates are known to be both toxic and carcinogenic. The results of this investigation could provide direction in the development of environmentally safe coatings.

STATUS OF EFFORT

A study has been initiated to investigate the effects of three parameters: type of inhibitor present in primer coating, presence or absence of a biocide in primer coating, and inoculation with microorganisms, on the extent of corrosion of coated aluminum panels. Three variations of inhibitor are being studied, chromate inhibitor, a non-chromate inhibitor, and no inhibitor. The study is also designed to investigate three microorganism inoculations: fungal consortium, bacterial consortium and sterile.

The study began on May 4, 1998, thus only 12 weeks exposure data has been obtained. Comparison of panels at this early stage indicates the presence of a biocide may reduce corrosion. There is also indication that panels inoculated with a bacterial consortium show more corrosion than those inoculated with a fungal consortium. However these early findings are based on very limited data and no conclusions should be drawn at this point.

During this reporting period we have also made substantial progress toward modifying an instrumentation design that will allow micro study of biodegradation and corrosion mechanisms. A Quartz Electrochemical Micro Balance (QEMB) system has been designed and six systems have been constructed and calibration. These units will be interfaced with a data acquisition system to monitor reactions and study degradation mechanisms of polymeric coatings in a variety of biological environments. This work will be an extension of the macro work currently underway and described above .

[1] Thorp, K., A. Crasto, J.-D Gu, R. Mitchell, "Contribution of Microorganisms to Corrosion," CORROSION/97, Paper No. 207, NACE International, Houston, TX (1997).

ACCOMPLISHMENTS/NEW FINDINGS

Macro Studies

To study the effects of inhibitors and biocides on corrosion activity, five polyamide-based primer coatings were obtained from DEFT, Inc. with the characteristics shown in Table 1. To investigate the premise that corrosion activity is enhanced by biological activity each of the five coatings is being tested under three conditioning environments: inoculation in a bacteria consortium, inoculation in a fungal consortium, and inoculation in a sterile media. Researches at the Laboratory of Microbial Ecology at Harvard University provided the bacterial and fungal consortium. These consortium were isolated as part of there ongoing research in the field of biocorrosion. The coatings were applied to clad 7075 aluminum panels measuring 7.6 cm by 15.2 cm.

Table 1. Characteristics of Primer Coatings for Corrosion Study

	Biocide Present	Biocide Absent
Chromate Inhibitor Present		X
Non-Chromate Inhibitor Present	X	X
Inhibitor Absent	X	X

Corrosion testing is being performed in accordance with ASTM D2803-82 using Procedure A. All panels were cleaned with an acid wash procedure, coated with primer using a standard spray technique, and scribed with a diamond-tipped stylist. For each of the five coatings, 24 panels were then inoculated with bacteria consortium in a sustaining broth, 24 panels inoculated with fungal consortium in a sustaining broth, and 9 panels sterilized and dipped in a sterile sustaining broth. All panels were then exposed, in batches, to a salt fog for 24 hours and conditioned at 25°C and 85% relative humidity in separate chambers, corresponding to the three exposure environments. The test was initiated the week of May 4, 1998. The study is designed to last a total of 36 weeks.

Panels have been pulled for analysis every four weeks since the initiation of the study. Corrosion development has been documented from visual observation. Upon removal from the environmental chamber, panels were washed with deionized water, and a standard tape-peel test was performed. A numerical ranking was then given for each of four visual observations: percent of scribed site effected by corrosion (site), length of average filiform corrosion (length), extent of pitting in unscribed areas (pitting), and amount of coating removed in tape-peel test (tape). In all rankings a 0 corresponds to no sign of corrosion while a ranking of 5 corresponds to 100% corrosion. In addition to the visual observations, images of the scribed area of the panels were obtained using a light microscope and associated image analysis system.

The average rankings for each of the four observations as a function of coating for panels conditioned 12 weeks are shown in Figure 1. Rankings of extent of corrosion as a function of

conditioning environment at 12 weeks are shown in Figure 2. This is very early data and it is not necessarily expected that panels exposed for longer duration will display the same trends as are seen in this 12 week data.

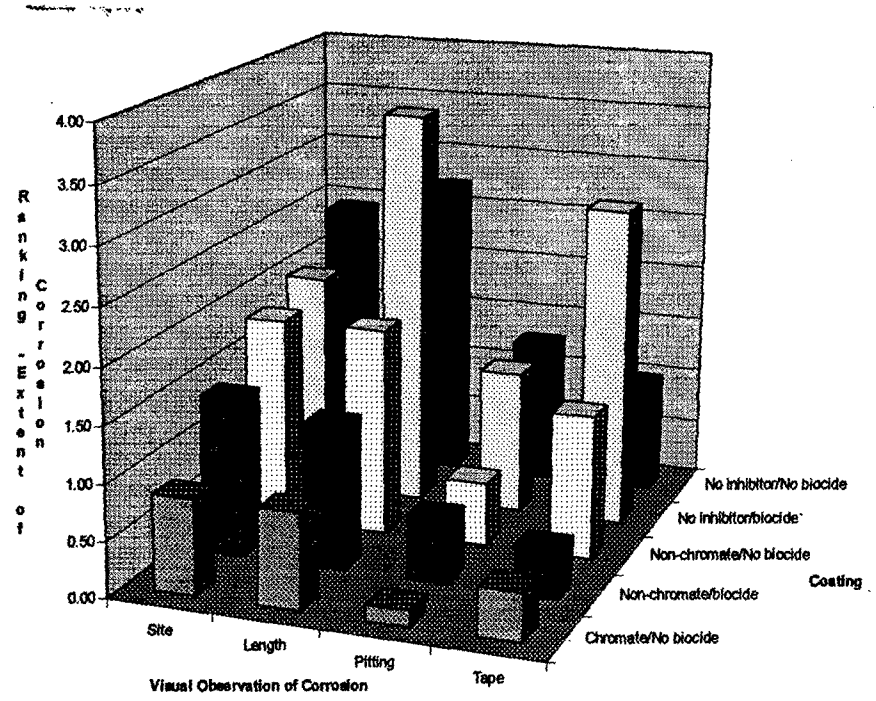

Figure 1. Characterization of corrosion activity as function of coating at 12 weeks

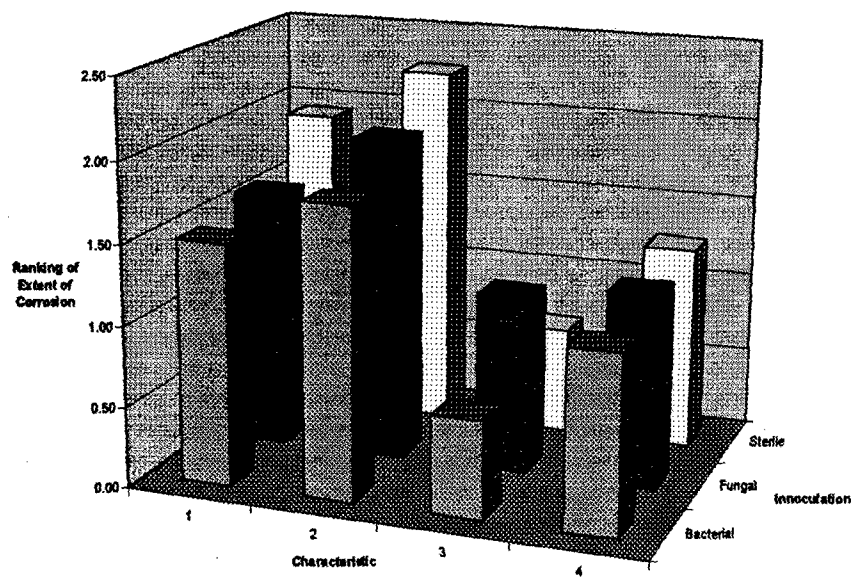

Figure 2. Characterization of corrosion activity as function of conditioning environment at 12 weeks

Micro Studies

Quartz Electrochemical Micro Balance (QEMB) systems have been designed for use in the study of mechanisms of biodegradation of polymeric coatings. The systems will permit the application of a coating directly onto a quartz crystal. The crystal can be placed in a controlled biological/chemical environmental and the frequency of the vibration of the crystal monitored. The frequency of the coated crystal will vary as degradation occurs and thus information can be obtained concerning the kinetics of the biodegradation activity. The controlled environment and design of the QEMB will also allow for high resolution study of the surface of the coated crystal.

A total of six modified QEMB systems have been constructed and calibrated. As shown, the frequency of the crystal vibration is directly proportional to the microbalance output voltage. A detailed description of the working of the circuit follows.

QEMB Circuit Description

The circuit of the QEMB is designed to (1) find the frequency difference between two quartz crystal oscillators, (2) translate this difference from a TTL circuit to an analog equivalent while physically separating the two sections, and (3) convert the frequency wave to a voltage which can be read into a data acquisition device. The circuit can be divided into three main sections: (1) the digital oscillator circuit combined with the TTL representation of the frequency difference, (2) the analog optical isolator designed to separate the digital section of the circuit from the analog section, and (3) the frequency to voltage converter combined with the data acquisition device.

The digital section of the circuit can be broken into two smaller sections: (1) the two oscillator circuits and (2) the calculation of the frequency difference. At the beginning of the circuit, since there are two distinct oscillators, there are two distinct oscillator circuits. The purpose of these circuits is to maintain the natural resonance frequency of 5 MHz through the use of both RC coupling and time constants, and the natural desire of the 74LS04 Hex Inverter, when power is applied, to immediately convert any lows to highs. Once power is applied to the IC, since there in nothing else providing power, the inputs to each of the inverters is low. Therefore, on the outputs, there will immediately be a TTL high, and resonance circuit is begun. Due to the RC coupling in each system, the resonance of the circuit will be dictated by the time constant of the RC system and by the natural frequency of the oscillator. The RC system provides the basis for the normal mode of the oscillator. In the working crystal, when there is a changing frequency, the inductor serves to provide an accurate normal mode as dictated by the new frequency of the oscillator.

When the signal exits the oscillator circuit, it immediately passes through another inverter as a precursor to passing through the Schmitt Trigger inverter on the 74LS14. The Schmitt Trigger is used to provide greater signal switching stability to take into account the possibility of an unclean signal inherent in a non-ideal system. After the signals pass through the inverters, they immediately enter a dual D flip-flop system (the 74LS74), with the reference signal going into the clock setting of the first flip-flop, and the working signal going into the D setting of the flip-flop. This configuration passes the value of the

working signal when the reference signal is high, therefore providing as output the absolute frequency difference between the two oscillators. The second flip-flop is simply used as an output buffer, passing the same signal output as the first, but providing greater output stability than can be achieved using only one flip-flop.

The output from the flip–flop system is then passed into the second section of the circuit, the TLP-2630 optical isolator. The optical isolator is comprised of two primary components: (1) a photodiode designed to emit light when the signal passes in only one direction, and (2) a detector physically separated from the photodiode to detect the light output and to translate it to an analog signal. The digital section of the circuit is physically separate from the remainder of the circuit. In order to guarantee this, the power, specifically the ground, to the digital ICs and photodiode of the TLP-2630, is different than that provided to the remainder of the circuit. This prevents ground loops in electrochemical experiments by physically separating the ground of the oscillator system from the ground of the recording hardware following the optical isolator. The power supply and ground are provided to the first half of the circuit from a 9V battery dropped to 5V through the use of the LM78L05 voltage converter. The remainder of the circuit is powered by a wall supply with outputs for +5V, GND, +12V, and –12V.

The output of the optical isolator is fed into the input of the LM2917 frequency to voltage converter. The internal components of the LM2917 use an operational amplifier comparator to ground to determine the input frequency, but the signal from the TLP2630 is a square wave oscillating from approximately 0.5V to 5.0V, thus never crossing ground. If this signal is fed, unadulterated, into the LM2917, no signal will be detected, and no output will be recorded. Therefore, an adding op amp circuit was constructed adding approximately –3.0 V to the signal, thus providing an output signal oscillating between about –2.5V and +2.0V, perfect for input into the LM2917. Due to capacitance inherent in the feedback resistor, the signal is also integrated, providing a triangle wave output. The new waveform is used as input into the LM2917 for frequency to voltage conversion. The voltage output of the LM2917 is linear with respect to the input frequency. Calibration curves were created, allowing data analysis using an ADC interface.

PERSONNEL SUPPORTED

Roger L. Vissoc	Professional Engineering Technologist
Kimberly A. Trick	Assistant Professor
R. Gerald Keil	Professor of Chemistry
Robert Dollinger	Undergraduate Student

PUBLICATIONS

None at this date.

INTERACTION/TRANSITIONS

Researchers on this program have coordinated activities with the Laboratory of Microbial Ecology at Harvard University. This interaction directly influenced the decision to study the effect of both the fungal and bacterial environments and has also allowed for the sharing of materials. Both of which have enhanced the validity of the research conducted. The interaction has also ensured the use of appropriate methods and analysis techniques for dealing with the microbial species. In addition, all research activities have been reviewed by and coordinated with researchers at the Air Force Wright Laboratory Materials Directorate to insure appropriate experimental conditions and corrosion testing.

NEW DISCOVERIES, INVENTIONS, PATENT DISCLOSURES

None at this date.

HONORS/AWARDS

None at this date.